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FINAL REPORT P-3058/C-2

Exhaust Particulate Filter Performance Study

to
CANMET
Energy, Mines and Resources
555 Booth Street
Ottawa, Ontario

K.E. Chipps
Dept. of Environmental Chemistry

March 26, 1980

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Exhaust Particulate Filter Performance Study

(Proposal P-3058/G)

to

CANMET

Energy, Mines and Resources

555 Booth Street

Ottawa, Ontario

1. SUMMARY

The objective of this study was to test the Canmet Filter for its performance as an exhaust particulate filter, and also to study its operational characteristics so that an assessment of its potential could be made.

Particulate removal efficiency of the filter averaged 42% with a range of 23%. This wide range of results was due to various filter modifications and alterations made throughout the testing program.

Leakage of unfiltered exhaust gases into the outlet of the filter was a major problem. This problem could not be controlled, therefore the results obtained cannot be used as a measure of the filtering efficiency of the filter cloth.

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March 26, 1980

2. INTRODUCTION

Testing of the Canmet Filter was done in two stages.

In the first stage, the total filter, with thirteen filter sections operational, was tested. Test results were not as expected, so a second stage of testing was performed.

During this second stage of testing, only six of the thirteen filter sections were left operational. Other alterations tested in this second stage were the injection of water to cool the exhaust prior to entering the filter, and removal of the filter sides to help prevent reentrainment of unfiltered exhaust into the outlet of the filter.

3. PARTICULATE SAMPLING TECHNIQUE

Particulate sampling of the engine exhaust was carried out using the E.P.A. Method 5 sampling technique (11) with a train manufactured by Joy Manufacturing. (see Figure 1).

The undiluted gases were sampled isokinetically and drawn through a short, heated, stainless steel probe to a 125 mm diameter Pallflex filter located in an oven maintained at > 120°C. The filtered gases were then bubbled through 250 ml of distilled water, divided between two Greenberg-Smith impingers sitting in an ice bath, dried with silica gel and delivered to a control module through an umbilical cord. The control module contains the diaphragm pump, gas meter, calibrated orifice, temperature monitors and controls.

The Pallflex T60A20 filters were conditioned prior to all weighings in a conditioning room maintained at 23°C and 50% R.H.

After each test, the probe and nozzle were rinsed with a 50% isopropyl alcohol and cyclohexane mixture, and a brush was passed through the probe to ensure the complete collection of particulate matter deposited in it. In the laboratory, the probe rinse was filtered to obtain an insoluble weight, and the filtrate was evaporated on a steam bath to determine the organic condensible portion of the probe rinse.

The total filter particulate was weighed and the filter extracted with cyclohexane in a Soxhlet apparatus to remove any soluble matter present on the filter. It was assumed that all organic matter was removed by this technique, as past tests have shown that no additional weight loss occcurred when the filters were re-extracted with dichloromethane. The amount of organic matter found on the filter depended on the filter temperature, which varied from test to test due to the exhaust gas, or sample, temperature. The impinger solutions were extracted with freon (Standard Methods for the Examination of Water and Wastewater, Method 502A) to measure any organic material which was condensed downstream of the filter. Therefore, condensible organic matter, as discussed in this report, comprises probe rinse and impinger condensibles and filter cyclohexane extractables. The cyclohexane insoluble matter, which is essentially carbon, consists of the particulate portion of the probe rinse and the filter cyclohexane insoluble matter. Thus, total particulate matter is the sum of the insoluble and organic portions.

4. TESTING OF TOTAL FILTER

Before commencing with the test program, filter sides were fabricated and installed (Figure 3). This was done to help control exhaust leakage from the filter, and also prevent the possibility of diluting the exhaust with outside air due to localized low pressure areas within the filter.

The first set of tests was performed using the total filter which was comprised of thirteen filter sections. Three different load speeds were tested. The results of these tests are shown in Table 1.

With all thirteen filter sections operational, the highest pressure drop recorded across the filter was 20 cm. of H_2O. This was at maximum load speed and after eleven hours of engine running time. Because a higher pressure drop across the filter was desired, the decision to modify the filter before further testing was made.

5. TESTING OF VARIOUS FILTER MODIFICATIONS

To increase the pressure drop across the filter, a portion of the filter was blocked off leaving only six of the original thirteen filter sections operational.

Fresh filter material was also installed prior to the second stage of testing. Special care was taken to install the filter material in a fashion which would limit, as much as possible, both exhaust leakage and reentrainment of unfiltered exhaust into the outlet of the filter.

The first four tests in Table 2 show the results obtained with the above filter modifications. A drop in filter efficiency of approximately eight percent occurred. There are two possible reasons for this decrease in efficiency:

(i) the velocity of the exhaust gas through the filter material was increased, and residence time in the filter material decreased by a factor of 2.2.

(ii) the increased pressure in the filter when partially blocked off created more exhaust leakage and more reentrainment of unfiltered exhaust into the outlet of the filter.

Two tests were performed to observe the effect of cooling the exhaust gas prior to its entering the filter.

An atomized water spray was used and the exhaust temperature was reduced to around $100^{\circ}C$. Results of these tests, are seen in Table 2, show a decrease in filter efficiency of 11% as compared to similar tests with no exhaust cooling. The probable reason for this decrease was that water droplets were impinging on the surface of the filter material and running down the material, through the filter frames and dripping into the outlet of the filter, while carrying with it carbon black which had been collected on the filter surface.

One other filter alteration was tested. This was the running of two particulate tests with the filter sides removed. The rationale behind these tests was that with the sides removed, any exhaust leakage through the sides of the filter would disperse into the ambient air thereby preventing reentrainment.

However, this did not prevent unfiltered exhaust gas from leaking through the bottom edges of the filter frames into the filter outlet.

Results of the tests with the filter sides removed are given in Table 2. A filter efficiency decrease of 9% was observed as compared to similar test conditions with the sides installed.

The exhaust flowrate downstream of the filter outlet was measured both with and without the filter sides installed. With the filter sides installed, the measured exhaust flowrate was essentially the same as the theoretical exhaust flowrate. ($5.40\ m^3 - min.^{-1}$ (dry std.). With the filter sides removed, the exhaust flow dropped to $1.84\ m^3 - min.^{-1}$ (dry std.). This represents a leakage of sixty six percent of the total exhaust through the sides of the filter.

6. CONCLUSIONS

Exhaust leakage through cracks between the filter cloth and the edges of the filter frames was the major problem encountered with the filter. Poorly finished welds, and warpage of the filter frames prevented the filter from fitting together tightly.

Because of the exhaust leakage problem, and the amount of unfiltered exhaust gas passing through to the filter outlet, it is impossible to estimate the potential collection efficiency of the filter cloth.

The cleaning or replacement of the filter cloth is a time consuming, laborious task. The disassembly and reassembly procedures, the time involved in cutting separate pieces of filter material for each frame, and the care necessary in replacing each individual frame with the fresh filter cloth intact, make the cleaning process difficult.

Life of the filter between cleanups was good. With the full filter operational, the pressure drop across the filter increased by 2 cm. of H_2O over 12.5 hours of engine running time. The pressure drop across the filter increased by 5 cm. of H_2O over 30 hours of running when only six of the total thirteen filter sections were left operational.

However, if exhaust leakage was eliminated, the filter collection efficiency would increase and the carbon buildup on the filter cloth would increase more rapidly, necessitating the cleanup operation at more frequent intervals.

FIGURE I

SAMPLING TRAIN

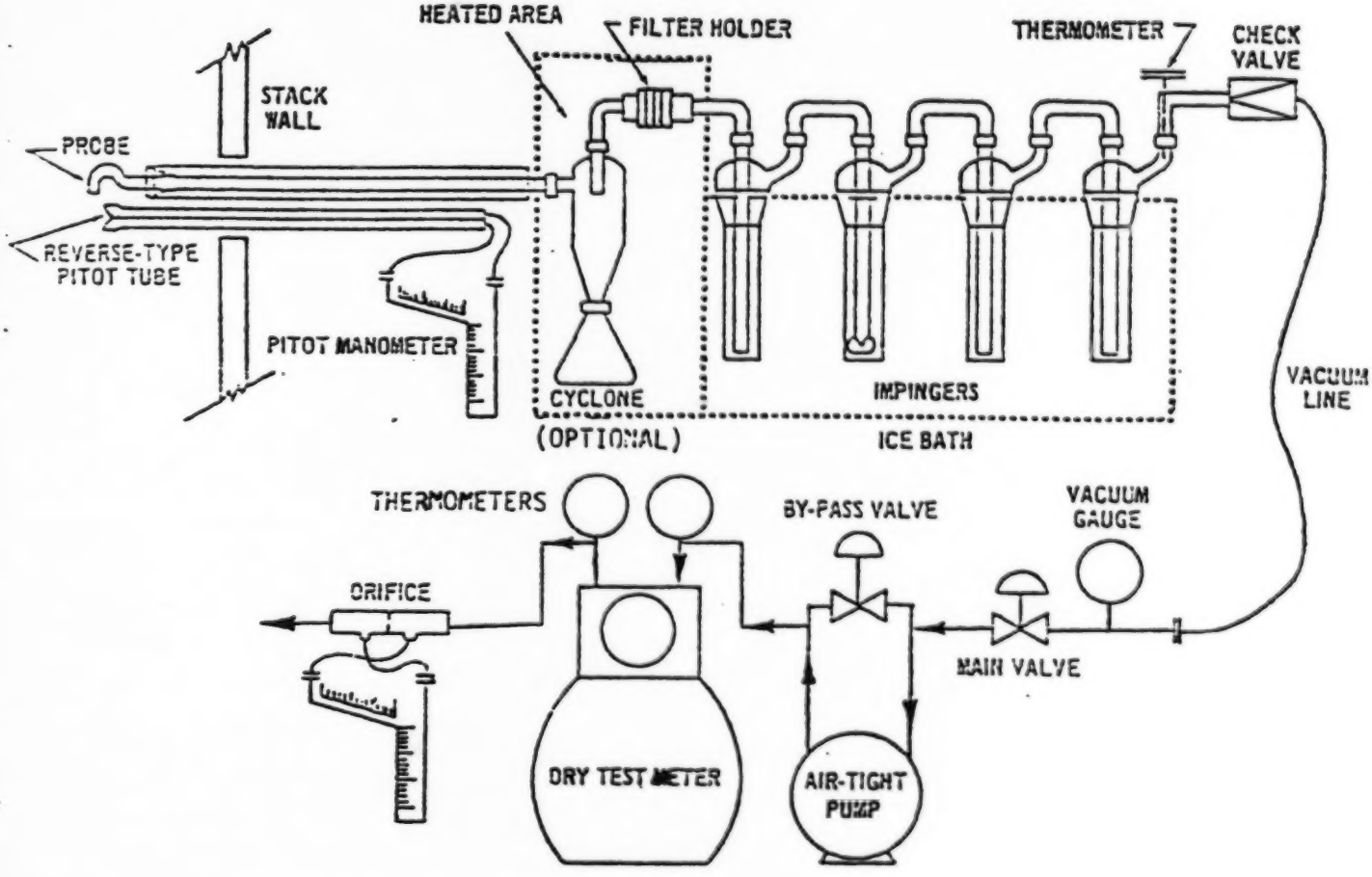


FIGURE 2

CANMET FILTER INSTALLATION

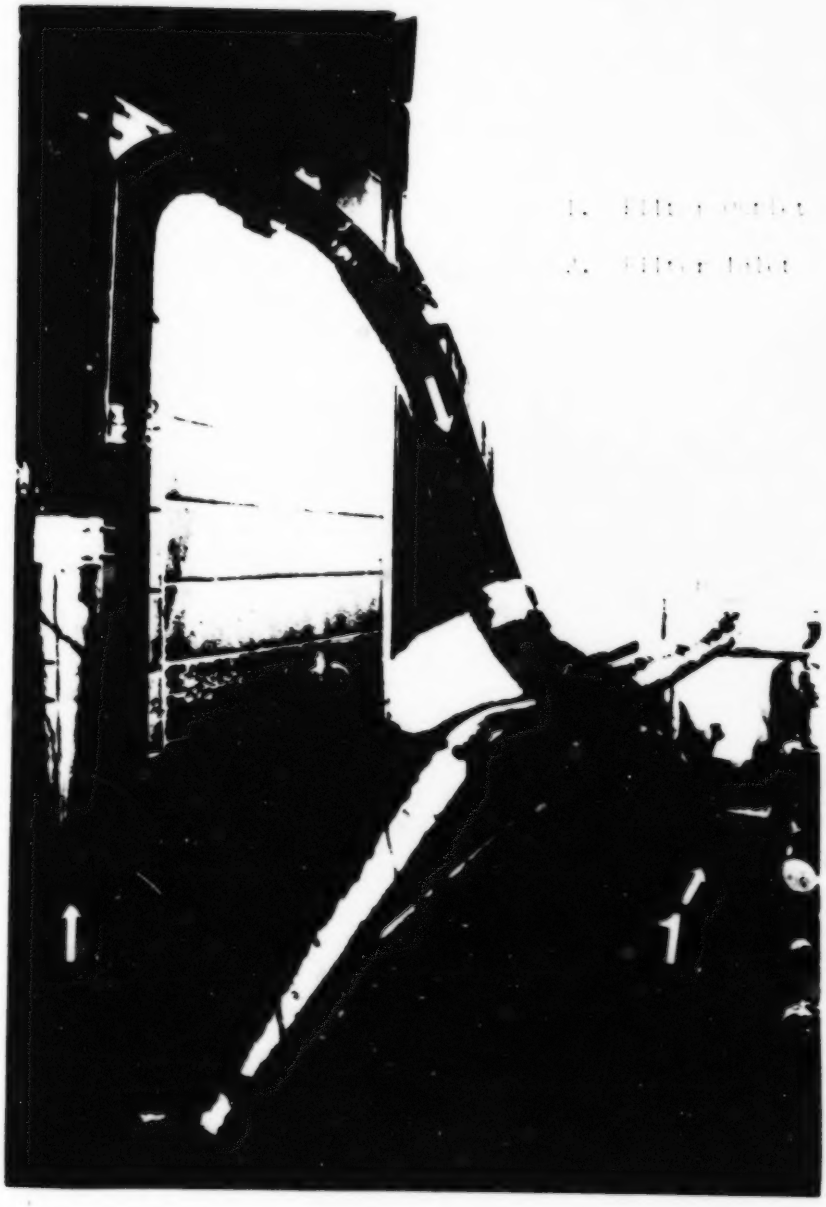
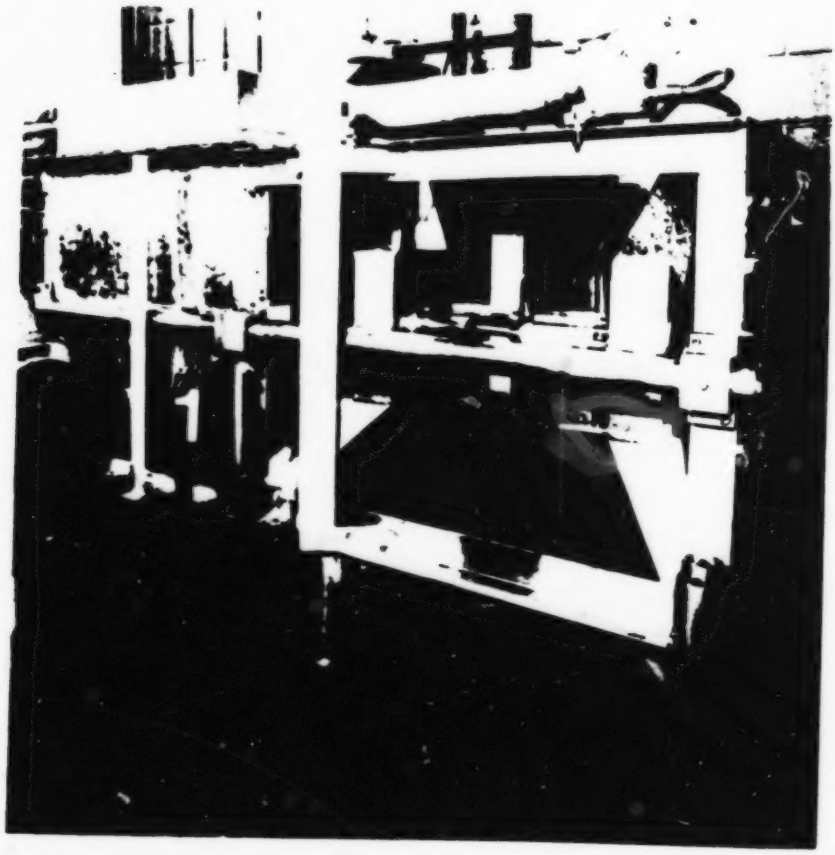


FIGURE 3

CANMET FILTER WITH SIDES INSTALLED



1. Filter Sides fabricated to prevent exhaust leakage.

TABLE I

Particulate Removal Capabilities of the Canmet Filter with the Total Filter Operational. (13 Filter Sections)

Speed (rpm)	Load (kW)	Torque (N.m)	Theoretical Exhaust Flowrate (m^3-min^{-1}) dry std.	Hrs. of Running Prior to Test	Filter Inlet Temp. (°C)	Engine Back Pressure (cm H_2O)	ΔP Across Filter (cm H_2O)	Location wrt Filter	Particulate Concentrations and Emission Rates					
									Total Particulate			CH Insoluble Particulate		
									mg/m^3	g/hr	% eff.	mg/m^3	g/hr	% eff.
1500	64	407	5.40	1.5	288	21	11	INLET OUTLET	84.7 47.8	27.5 15.5	44	75.4 36.4	24.5 11.8	52
1500	64	407	5.40	3.5	288	23	12	INLET OUTLET	80.2 46.5	26.1 15.1	42	75.3 38.8	24.5 12.6	48
1850	78	407	6.49	5.5	340	27	15	INLET OUTLET	69.0 40.6	26.9 15.9	41	66.6 34.4	26.0 13.4	48
1850	78	407	6.49	11.0	340	28	16	INLET OUTLET	66.0 34.5	25.8 14.5	48	63.5 30.4	24.8 11.9	52
2200	93	407	7.46	7.0	380	36	20	INLET OUTLET	65.2 43.5	29.3 19.5	33	60.7 34.6	27.3 15.	43
2200	93	407	7.46	9.0	380	36	20	INLET OUTLET	56.6 30.0	25.4 13.5	47	52.6 24.5	23.6 11.0	53

TABLE 2

Particulate Removal Capabilities of the Canmet Filter With Only Part of Filter Operational (6 Filter Sections)

Hrs. of Running Prior To Testing	Test Description	Engine Back-Pressure (cm H_2O)	ΔP Across Filter (cm H_2O)	Location wrt. Filter	Particulate Concentrations and Emission Rates					
					Total Particulate			CH Insoluble Particulate		
					mg/m^3	g/hr	% eff.	mg/m^3	g/hr	% eff.
10.5	Hot Filter (278°C)	27	17	Inlet Outlet	89.7 57.5	29.1 18.7	36	86.2 53.0	28.0 17.2	39
13.0	Hot Filter (267°C)	27	17	Inlet Outlet	76.3 44.8	24.8 14.6	41	71.0 38.4	23.1 12.5	46
15.0	Hot Filter (275°C)	28	18	Inlet Outlet	92.0 54.6	29.9 17.7	41	86.8 49.4	28.2 16.1	43
23.0	Hot Filter (276°C)	30	17	Inlet Outlet	106.4 67.9	34.6 22.0	36	104.8 63.7	34.0 20.7	39
17.0	Pre-Filter Exhaust Cooling (103°C)	24	14	Inlet Outlet	72.8 53.6	23.6 17.4	26	67.4 47.4	21.9 15.4	30
18.5	Pre-Filter Exhaust Cooling (100°C)	25	15	Inlet Outlet	63.9 43.7	20.8 14.2	32	57.4 39.2	18.6 12.7	32
27.0	Hot Filter Sides Removed	28	18	Inlet Outlet	110.6 75.7	35.9 24.6	32	108.7 71.9	35.3 23.3	34
28.5	Hot Filter Sides Removed	28	19	Inlet Outlet	110.3 76.5	35.8 24.8	31	104.5 71.8	33.9 23.3	31

Speed = 1500 rpm
Load = 64 KW
Torque = 407 N-M

FINAL REPORT P-3058/G-1

Smokemeter Correlation Study
to
CANMET
Energy, Mines and Resources Canada,
555 Booth Street,
Ottawa, Ontario.

K.E. Chipps,
Dept. of Environmental Chemistry.

July 18, 1979

ONTARIO RESEARCH FOUNDATION
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Smokemeter Correlation Study

(Proposal P-3058/G)

to

CANMET

Energy, Mines and Resources Canada

555 Booth Street,

Ottawa; Ontario.

1. SUMMARY

A correlation study of the opacity readings from a Celesco Model 107 diesel exhaust smokemeter with gravimetric particulate concentrations was performed on the exhaust of an indirect injection Deutz F6L-714 diesel engine.

From the data obtained, a linear correlation appears to exist between the smokemeter opacity and the cyclohexane insoluble particulate concentration. However, the calibration curve of the indirect injection Deutz engine did not correspond to the curve drawn by Canmet for a Ford direct injection diesel engine.

It appears that this smokemeter could be used as an alternative to time consuming gravimetric tests, with the following reservations:

(i) There would be no way of distinguishing how much hydrocarbon material is present in the exhaust stream.

(ii) The calibration curve may vary with hydrocarbon concentration if this is a significant component of the total particulate. (Not significant in the case of the Deutz engine).

(iii) Calibration curves must be generated for each engine series to be tested.

Keith E. Chipps

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July 18, 1979

2. INTRODUCTION

The Canadian Explosive Atmospheres Laboratory has purchased and tested a Celesco Model 107 in-line diesel exhaust smokemeter. In their investigation of the correlation of smokemeter readings with exhaust particulate concentrations, it was concluded that the smokemeter output agreed extremely well with the gravimetric determination of smoke.

The above tests, however, were performed on a direct-injected diesel engine having particulate emissions up to 135 mg/actual m^3. It was decided, therefore, to attempt a similar correlation using a much 'cleaner' engine, such as the indirect-injection Deutz F6L-714 presently installed on a test bed at ORF. The results of such testing should offer some clarification in a decision to use the smokemeter, instead of time-consuming gravimetric tests, to measure diesel exhaust particulate matter.[2]

3. SMOKEMETER INSTALLATION

The smokemeter was installed according to the instruction manual provided. In order to simplify the installation, and to minimize alterations in the existing exhaust system, the sensor unit was inserted, vertically oriented, into the exhaust in the previous location of the venturi scrubber. A photograph of the installation is provided (see Figure 2).

Due to the extremely warm ambient temperatures during the testing program, the Haake constant temperature controller was not capable of maintaining the water temperature at the initially used 35°C.

Therefore, except for the three initial tests performed in March, all other tests were performed with a water bath temperature setting of 50°C.

Chromel-alumel thermocouples were used to monitor the temperature of the exhaust gas both upstream and downstream of the detector, as well as the cooling water in the feed and return hoses.

4. TESTING PROGRAM

The major problem encountered in the testing program was to get smokemeter opacity readings which were high enough to generate a meaningful calibration curve. With the engine set at standard injection timing (24°BTDC), the load speed of highest particulate emission was 1625 rpm and 80 kw. At this load speed, the corrected opacity reading was 2.9%.

To overcome this problem, the injection timing was advanced to 30°BTDC. This change increased the smokemeter opacity reading to 10.3% at 1625 rpm and 80 kw; an increase of 3.6 times the original.

Five load speeds were tested using 24°BTDC injection timing and five load speeds were tested using an injection timing of 30° BTDC. The data obtained from the 24° setting covered the lower range of the calibration curve while the data from the 30° setting covered the upper range of the calibration curve.

Gaseous THC readings at each load speed were also requested by Canmet. Readings were taken on an IPM. RS5, heated flame ionization detector analyzer.

5. PARTICULATE SAMPLING TECHNIQUE

Particulate sampling of the engine exhaust was carried out using the E.P.A. Method 5 sampling technique (11) with a train manufactured by Joy Manufacturing. (see Figure I).

The undiluted gases were sampled isokinetically and drawn through a short, heated, stainless steel probe to a 125 mm diameter Pallflex filter located in an oven maintained at >120°C. The filtered gases were then bubbled through 250 ml of distilled water, divided between two Greenberg-Smith impingers sitting in an ice bath, dried with silica gel and delivered to a control module through an umbilical cord. The control module contains the diaphragm pump, gas meter, calibrated orifice, temperature monitors and controls.

The Pallflex T60A20 filters were conditioned prior to all weighings in a conditioning room maintained at 23°C and 50% R.H.

After each test, the probe and nozzle were rinsed with a 50% isopropyl alcohol and cyclohexane mixture, and a brush was passed through the probe to ensure the complete collection of particulate matter deposited in it. In the laboratory, the probe rinse was filtered to obtain an insoluble weight, and the filtrate was evaporated on a steam bath to determine the organic condensible portion of the probe rinse.

The total filter particulate was weighed and the filter extracted with cyclohexane in a Soxhlet apparatus to remove any soluble matter present on the filter. It was assumed that all organic matter was removed by this technique, as past tests have shown that no additional weight loss occurred when the filters were re-extracted with dichloromethane. The amount of organic matter found on the filter depended on the filter temperature, which varied from test to test due to the exhaust gas, or sample, temperature. The impinger solutions were extracted with freon (Standard Methods for the Examination of Water and Wastewater, Method 502A) to measure any organic material which was condensed downstream of the filter. Therefore, condensible organic matter, as discussed in this report, comprises probe rinse and impinger condensibles and filter cyclohexane extractables. The cyclohexane insoluble matter, which is essentially carbon, consists of the particulate portion of the probe rinse and the filter cyclohexane insoluble matter. Thus, total particulate matter is the sum of the insoluble and organic portions.

6. TEST RESULTS

Table 1 gives individual test results while Table 2 gives the averaged results of the upstream and downstream position tests at each load speed.

Figure 3 is a plot of smokemeter opacity vs cyclohexane insoluble gravimetric particulate concentrations.

The smokemeter opacity readings correlated very well with the gravimetric particulate concentrations. The curve appears to be a straight line which passes through the origin. This suggests that the smokemeter is not capable of detecting hydrocarbon material present in the exhaust gas stream.

Figure 4, which is a plot of smokemeter opacity vs total gravimetric particulate concentration further supports this speculation. From the graph, a smokemeter opacity reading of 0.0% corresponds to a total particulate concentration of approximately 8 mg/m^3. This 8 mg/m^3 would represent the condensible organic fraction of the total particulate concentration.

A comparison plot of a direct injection diesel engine (Canmet data)(1) and an indirect injection diesel engine (ORF data) can be seen in Figure 5. The slopes of the two curves are considerably different with the slope of the curve for the direct injection engine being $1.143 \times 10^{-3} m^3/mg$ and a slope of $0.871 \times 10^{-3} m^3/mg$ for the indirect injection engine. This represents a difference in slope of 23%.

The most likely explanation of this discrepancy in the slopes of the two curves is the difference in the nature of the particulate matter produced by the two different engines. Particle size, shape and density, and the amount of hydrocarbons and their physical state (gaseous or liquid droplets) are all variables which could make the smokemeter respond differently.

Gaseous total hydrocarbon concentrations in ppm were taken at each load speed tested. Results can be seen in Table 3.

7. CONCLUSIONS

The smokemeter opacity readings correlated extremely well with the measured gravimetric cyclohexane insoluble particulate concentrations at each of the load speeds tested. From the testing done, it appears that this smokemeter could be an acceptable alternative to time consuming gravimetric tests, with the following reservations:

(1) There would be no way of distinguishing how much hydrocarbon material is present in the exhaust gas stream.

(2) It would appear that different engines would have different calibration curves. Before a smokemeter could be reliably used, a curve for that particular engine series would need to be generated.

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Gravimetric Correlation of an In-Line Smokemeter on a
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Report MRP/MRL 77-125 (TR); Ottawa, Ont., November, 1977.

2. Simmons, E.W.
Smokemeter Correlation Study for Canmet,
Energy, Mines and Resources Canada.
Addendum to ORF Report P-2512/G-5.

FIGURE I

SAMPLING TRAIN

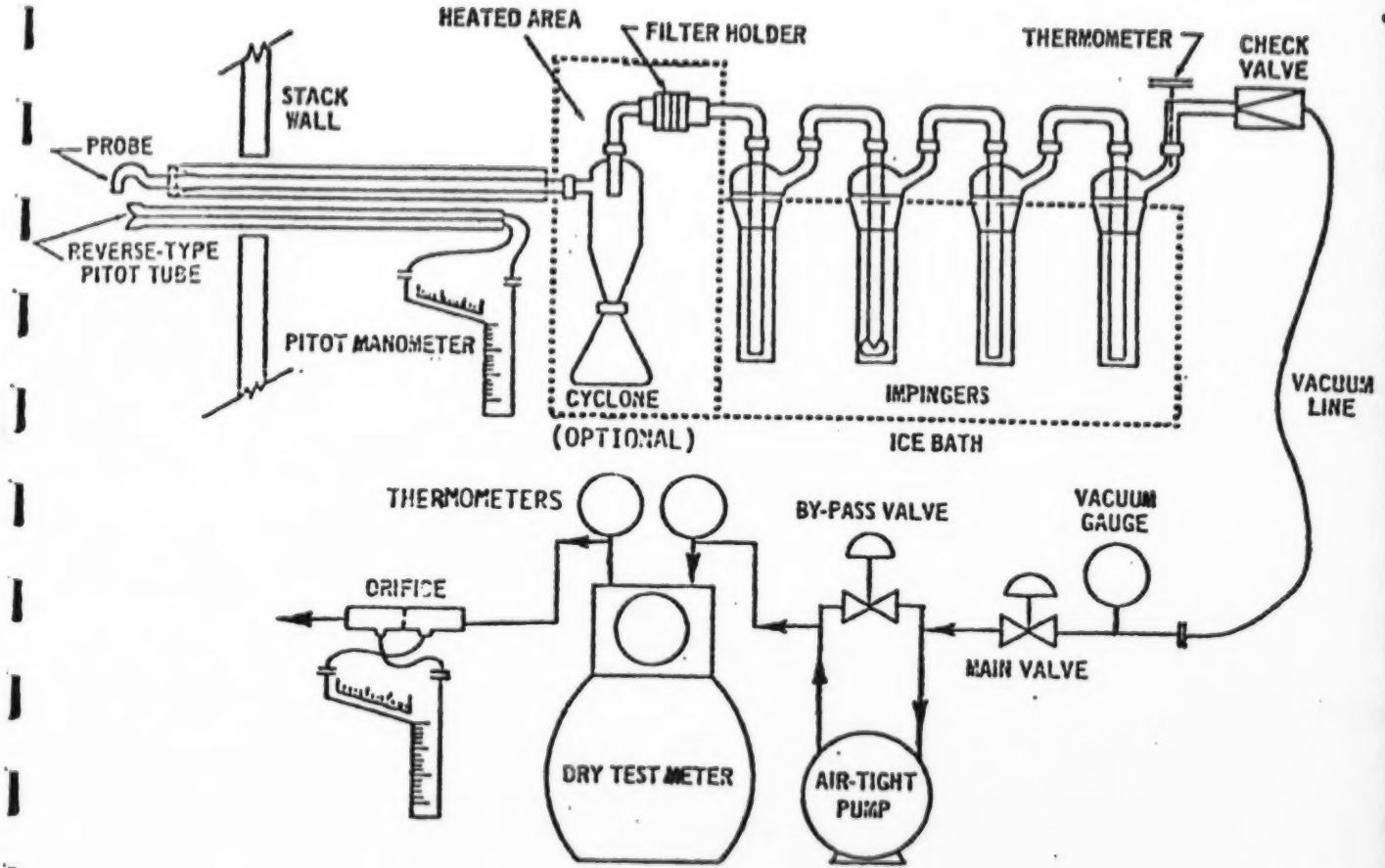

FIGURE 2

SMOKEMETER INSTALLATION

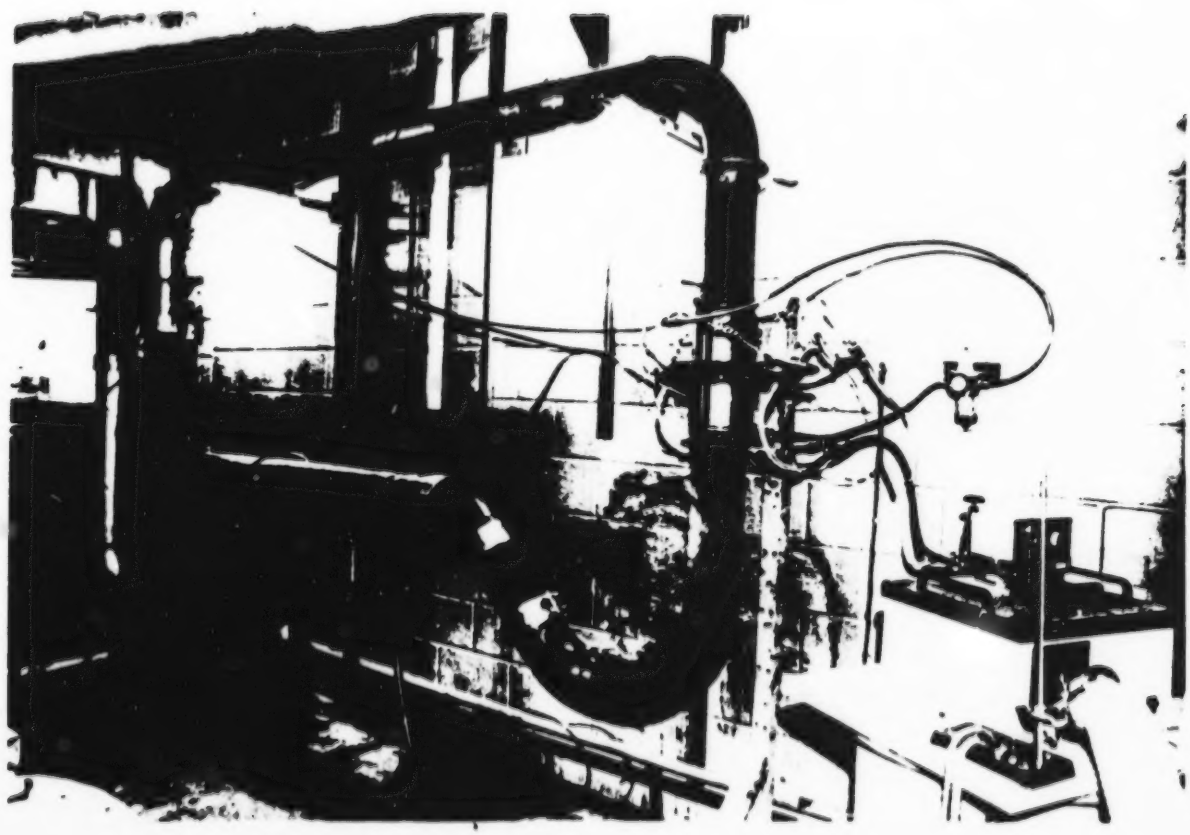

FIGURE 3

CH Insoluble Gravimetric Particulate Concentration
vs.
Smokemeter Opacity
Averaged Results

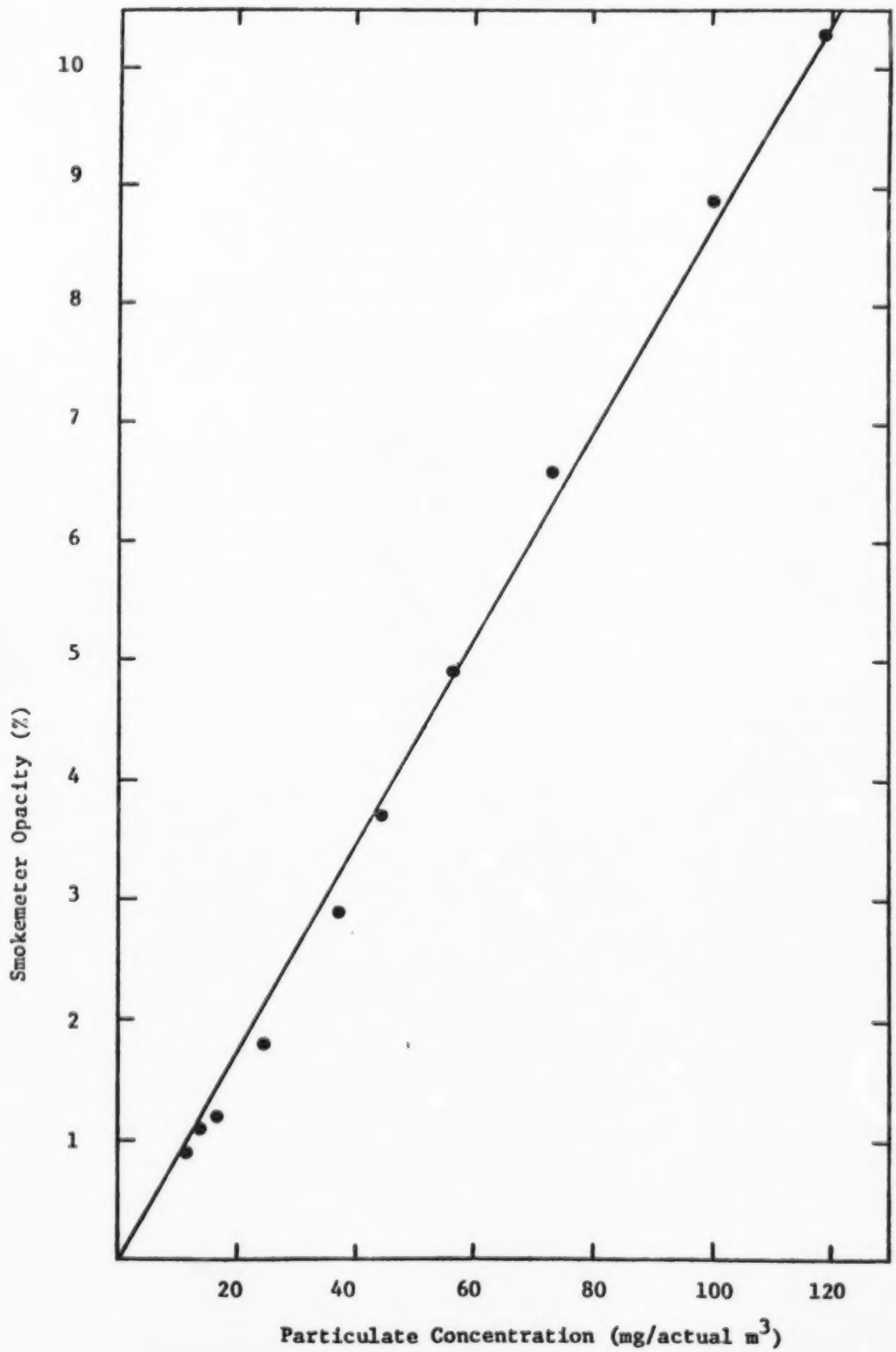

FIGURE 4

Total Gravimetric Particulate Concentration vs Smokemeter Opacity Averaged Results

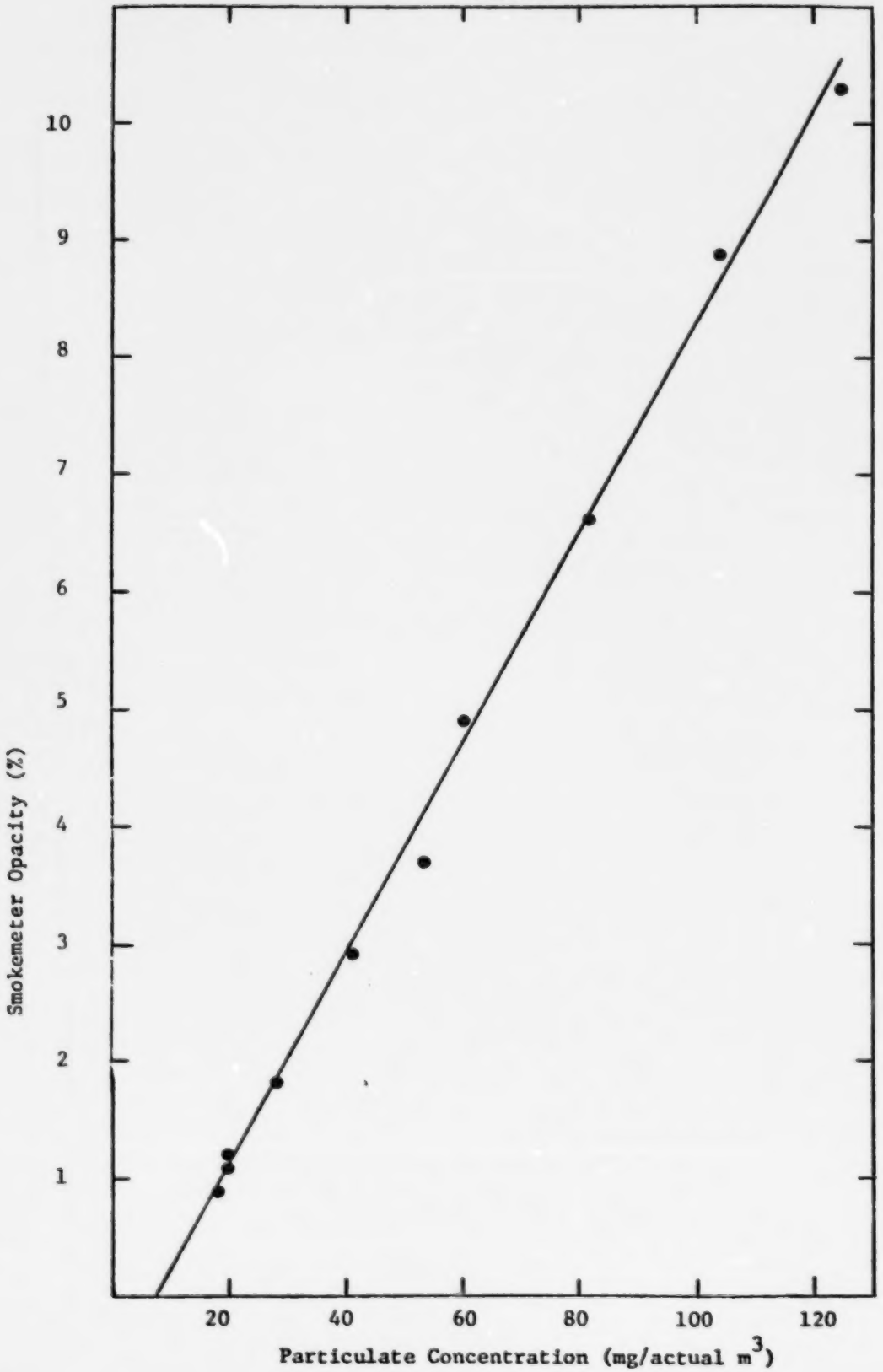

FIGURE 5

Particulate Concentration vs. Smokemeter Opacity Comparison of Canmet Data Using a Direct Injection Diesel Engine and ORF Data Using an Indirect Injection Diesel Engine

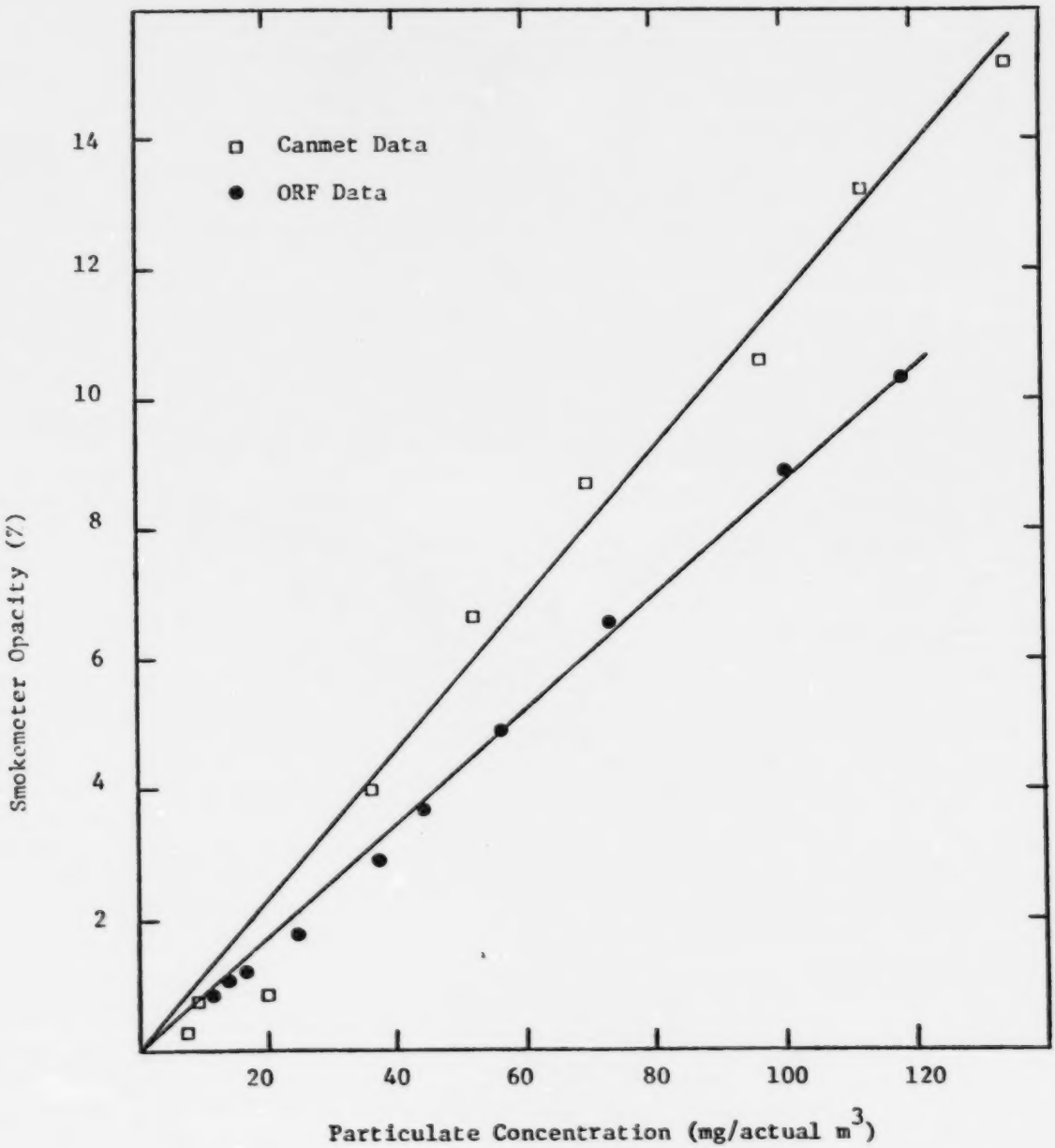

TABLE 1

Correlation of Smokemeter Opacity Readings with Gravimetric Particulate Concentrations. Individual Test Results

Sampling Position Relative to Smokemeter	Engine rpm	Power KW	Engine Torque N.m	Injection Timing ^{o} BTDC	Average Water Bath Temp. ^{o}C	Average Smokemeter Temp. ^{o}C	Opacity Reading %	Hot Zero Reading %	Corrected Opacity %	Total Gravimetric Particulate Conc.		CH Insoluble Gravimetric Particulate Conc.	
										mg/m^3	mg/actual m^3	mg/m^3	mg/actual m^3
Upstream	2000	57	270	24	35	248	0.4	- 0.5	0.9	34.3	19.7	22.6	13.0
Downstream	2000	57	270	24	35	248	0.3	- 0.6	0.9	29.1	16.7	17.1	9.8
Upstream	2000	28	135	24	35	181	0.8	- 0.3	1.1	29.9	19.6	21.1	13.8
Upstream	2200	93	405	24	49	376	0.5	- 0.7	1.2	46.6	21.5	40.4	18.7
Downstream	2200	93	405	24	49	376	0.5	- 0.7	1.2	38.0	17.5	32.6	15.1
Upstream	2000	85	406	24	35	348	1.1	- 0.7	1.8	62.5	30.1	57.1	27.5
Downstream	2000	85	406	24	35	348	1.0	- 0.8	1.8	54.2	26.1	47.2	22.7
Upstream	1625	80	469	24	51	333	2.7	- 0.2	2.9	83.0	40.7	71.7	35.2
Downstream	1625	80	469	24	51	333	2.7	- 0.2	2.9	86.8	42.6	80.5	39.5
Upstream	2200	71	307	30	50	301	3.4	- 0.3	3.7	111.5	58.0	91.4	47.6
Downstream	2200	71	307	30	50	301	3.4	- 0.3	3.7	93.2	48.5	78.7	41.0
Upstream	2200	101	437	30	55	394	5.4	+ 0.5	4.9	145.9	65.6	135.9	61.1
Downstream	2200	101	437	30	55	394	5.4	+ 0.5	4.9	122.5	55.1	113.5	51.0
Upstream	1500	63	403	30	50	286	6.1	- 0.5	6.6	170.3	90.4	150.2	79.8
Downstream	1500	63	403	30	50	286	6.1	- 0.5	6.6	138.6	73.6	125.7	66.8
Upstream	1625	76	447	30	52	333	8.5	- 0.4	8.9	231.2	113.1	220.7	108.0
Downstream	1625	76	447	30	52	333	8.5	- 0.4	8.9	193.2	94.5	189.3	92.6
Upstream	1625	80	469	30	52	333	10.6	+ 0.3	10.3	249.4	122.4	239.2	117.4
Downstream	1625	80	469	30	52	333	10.6	+ 0.3	10.3	257.6	126.4	245.0	120.2

TABLE 2

Correlation of Smokemeter Opacity Readings with Gravimetric Particulate Concentrations.

Averaged Results.

Engine rpm	Power KW	Engine Torque N.m	Injection Timing °BTDC	Average Water Bath Temp °C	Average Smokemeter Temp. °C	Opacity Reading %	Hot Zero Reading %	Corrected Opacity %	Total Gravimetric Particulate Conc.		CH Insoluble Gravimetric Particulate Conc.		Condensible Organic Material mg/actual m^3
									mg/m^3	mg/actual m^3	mg/m^3	mg/actual m^3	
2000	57	270	24	35	248	0.4	- 0.5	0.9	31.7	18.2	19.9	11.4	6.8
2000	28	135	24	35	181	0.8	- 0.3	1.1	29.9	19.6	21.1	13.8	5.8
2200	93	405	24	49	376	0.5	- 0.7	1.2	42.3	19.5	36.5	16.8	2.7
2000	85	406	24	35	348	1.1	- 0.7	1.8	58.4	28.1	52.2	25.0	3.1
1625	80	469	24	51	333	2.7	- 0.2	2.9	84.9	41.7	76.0	37.3	4.4
2200	71	307	30	50	301	3.4	- 0.3	3.7	102.4	53.3	85.0	44.2	9.1
2200	101	437	30	55	394	5.4	+ 0.5	4.9	134.2	60.4	124.7	56.1	4.3
1500	63	403	30	50	286	6.1	- 0.5	6.6	154.5	82.0	137.9	73.2	8.8
1625	76	447	30	52	333	8.5	- 0.4	8.9	212.2	103.8	205.1	100.4	3.4
1625	80	469	30	52	333	10.6	+ 0.3	10.3	253.5	124.4	242.1	118.8	5.6

Table 3

Total Hydrocarbon Concentrations (ppm) at Each Tested Load Speed as Measured with a Heated FID. THC Analyzer

Engine rpm	Power kW	Torque N.m	Injection Timing $^{o}_{BTDC}$	THC Concentration ppm
2200	93	405	24	30
1625	80	469	24	15
2200	71	307	30	85
2200	101	437	30	55
1500	63	403	30	21
1625	76	447	30	21
1625	80	469	30	18

DRAFT
FINAL REPORT P-3058/G-3

Catalytic Purifier Comparison Study

to

CANMET
Energy, Mines and Resources, Canada
555 Booth Street,
Ottawa, Ontario.

E. W. Simmons
W. Drummond
Dept. of Environmental Chemistry

April 16, 1980.

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Catalytic Purifier Comparison Study
(Proposal P-3058/G)

to

CANMET
Energy, Mines and Resources, Canada
555 Booth Street,
Ottawa, Ontario.

1. SUMMARY

A pair of Oxycat exhaust purifiers, initially containing new catalyst pellets, then used pellets, were installed in the exhaust system of a Deutz F6L-714 diesel engine, and their effects on exhaust emissions compared.

New and used catalyst pellets exhibit similar capabilities in the control of carbon monoxide and hydrocarbons. The formation of nitrogen dioxide tends to become less apparent with the used catalyst than with the new.

Both new and used catalyst pellets emit sulphates. However, until possible anomalies in the sulphate data can be reviewed, no firm comparisons can be made. An updated report, including the verified sulphate data, will be issued in the near future.

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EWS/WD/id
April 16, 1980

2. INTRODUCTION

Catalytic purifiers are used in the treatment of diesel exhaust in an underground environment, for the control of carbon monoxide and organic emissions.

Some of these oxidation catalysts, however, have, in the past, been observed to promote the oxidation of nitric oxide and sulphur dioxide, present in diesel exhaust, to nitrogen dioxide and sulphate.

The purpose of this study, therefore, was to determine the difference in any such effect produced by new and used catalyst material.

3. CATALYTIC PURIFIER INSTALLATION AND MODIFICATION

Because of the physical size of these purifiers, and the space constraints imposed by the geometry of the engine and exhaust system, each catalytic unit was mounted horizontally, immediately adjacent to its exhaust manifold (Figure 1). This proximity to the manifold ensured that the purifiers would be subjected to the highest practical temperature for any given engine load/speed, since there would be little opportunity for heat loss to occur upstream of their location. However, in being installed horizontally, the catalyst fill port was then rendered virtually useless for checking and maintaining the catalyst level. It was agreed, then, with the scientific officer, that a new, vertical, fill port should be welded into the centre of each purifier. The location of the old and new fill ports is indicated in Figure 2.

4. TEST PROGRAM

New catalyst pellets were obtained from Mechron Engineering Products Ltd. and the purifiers were filled and shaken gently until their capacity was reached. The filled units were then installed in the exhaust system in the manner previously described. Prior to testing the emissions, the engine was operated for a two-hour period at various loads and speeds, after which time the catalyst beds were checked for settling and topped up with new pellets.

After the completion of the emission tests described below, used catalyst pellets, obtained from Inco Ltd., Sudbury, were substituted for the new pellets and the tests were repeated, using indentical handling and test procedures.

4.1 Sulphate and Sulphur Dioxide Sampling and Analysis

Controlled Condensation Technique

This technique uses a temperature-controlled sample condenser (Figure 3) to lower the sample temperature to below the dew-point of sulphuric acid, while maintaining it above the dew-point of the water contained in the exhaust. A condenser temperature of 70°C achieves this for the conditions which exist in diesel exhaust.

A frit before the outlet prevents H_2SO_4 mist (condensed SO_3) from being carried through the condenser. The condenser outlet is then attached to the conventional impinger train. The first two impingers contain 3% hydrogen peroxide to catch the SO_2, the third is empty, and the fourth contains silica gel to remove any remaining moisture in the sample before the dry gas meter.

The sampling procedure is then quite similar to EPA Method 5, except that the sampling rate is not isokinetic due to the unusual probe design and the low flow rate obtained.

Because of the complicated configuration of the condenser, a simple method of flushing out the condensed sulphuric acid was devised. The condenser is inverted and its outlet joint is attached to a Buchner flask. Vacuum is applied to the flask and water is admitted to the condenser inlet. The water rises up the condenser coil, wetting all its surface, and is deposited in the Buchner flask. Any hydrocarbons condensed in the coil do not appear to be washed off the walls by water, thereby eliminating potential hydrocarbon interference in the analytical technique. The resulting solutions are analyzed using a standard turbidimetric technique. For the detailed analytical procedure, see Appendix 1.

These tests were, of necessity, performed downstream of the catalytic purifiers only, in the combined exhaust gas from both units.

4.2 Continuous Gaseous Sampling and Analysis

4.2.1 Analytical Instruments

The gaseous components of the exhaust were monitored using the following instrumentation:

Carbon Dioxide	- Horiba AlA-21 NDIR Ranges 5%, 12%
Carbon Monoxide	- Horiba AlA-21-AS NDIR Ranges 200 ppm, 1000 ppm
Oxides of Nitrogen	- Thermo-Electron 10A, chemiluminescent stainless steel NO_2 to NO converter Ranges 2.5, 5, 25, 100, 250, 1000, 2500, 10,000 ppm
Total Hydrocarbons	- IPM Ratfisch, RS5, Heated FID Ranges 10, 100, 1000, 10,000 ppm
Oxygen	- Taylor Servomex OA 273, Paramagnetic Ranges 5%, 25%, 100%

4.2.2 Sampling Technique

A continuous sample was extracted from the exhaust stream, cooled, dried and filtered using the sampling system described below, the schematic of which is shown in Figure 4.

Glass wool was wrapped loosely over evenly spaced holes in a 1/4" stainless steel probe to prevent heavy particulate deposits from contaminating the sampling line. This probe, which was sealed at one end, was installed across the diameter of the exhaust pipe, and sample drawn through it to the sample conditioning cart through a 35-foot stainless steel tube maintained at 180°C. A portion of the hot sample was diverted to the total hydrocarbon analyzer by its own heated pump. The remaining sample passed through a cooling coil installed in an ice bath. Condensate was continuously pumped from the bowl of a Balston filter unit, through which the sample passed from the outside of the filter to the inside. This

kept the filter medium dryer, extending filter change intervals. Cool, dry and clean sample was pumped, using a stainless steel bellows pump, through a manifold to the relevant instruments. The appropriate flow to each analyzer was monitored with a rotameter. All concentrations, with the exception of total hydrocarbons, were measured on a dry basis.

It was originally planned to take these measurements in the exhaust immediately upstream of the purifiers by teeing the sampling line to each exhaust pipe, thereby drawing sample from both manifolds. The downstream samples would then have been taken from the combined exhaust of the two units. It then became apparent that the exhaust gas composition from each bank of cylinders was not the same, possibly due to backpressure differences. Since the sample drawn from the two exhaust pipes could not be guaranteed to give a representative exhaust gas composition under all conditions, it was decided to sample immediately upstream and downstream of one purifier only.

5. TEST RESULTS

It can be seen, from Table 1, that the ability of the catalyst to oxidize carbon monoxide and hydrocarbons appears to be little affected by use, although it has been learned that the used pellets have, at some time, been subjected to a reconditioning procedure in a gas-fired retort.

It would seem, however, that the new catalyst pellets are slightly more prone to produce nitrogen dioxide than the used pellets, under some conditions.

The formation and emission of sulphates will not be discussed in detail at this time, although the results, subject to verification, which are given in Table 1 tend to indicate that the storage and subsequent high temperature evolution of sulphates takes place.

6. CONCLUSIONS

- The ability of used catalyst pellets to control carbon monoxide and hydrocarbons is similar to that of new pellets.
- The emission of nitrogen dioxide shows a tendency to be somewhat lower from a purifier containing used catalyst pellets, than one containing new pellets.
- Sulphates are emitted from both new and used catalyst beds.

TABLE 1 - Emissions from Oxycat Purifiers
New Pellets vs Old

Speed RPM	Torque N.m.	Load kw	Catalyst	Inlet Temperature °C	ppm CO		ppm THC		ppm NO_2		ppm SO_4
					In	Out	In	Out	In	Out	
2200	404	93	New	612	210	32	48	6	10	10	11
			Used	622	190	47	48	15	0	0	80
2200	228	71	New	455	164	40	85	20	20	45	15
			Used	461	195	45	90	20	10	10	14
2200	149	47	New	334	180	54	90	34	25	25	2
			Used	341	140	74	62	35	20	20	3
1850	404	78	New	538	144	25	45	10	0	25	35
			Used	555	136	32	35	7	0	0	33
1850	228	60	New	396	108	36	48	16	30	40	10
			Used	408	108	32	42	20	30	40	6
1850	149	39	New	293	112	62	66	32	30	20	2
			Used	296	120	45	60	26	40	50	2
			New	481	104	.18	22	1	10	30	25
			Used	501	112	22	17	3	30	30	27

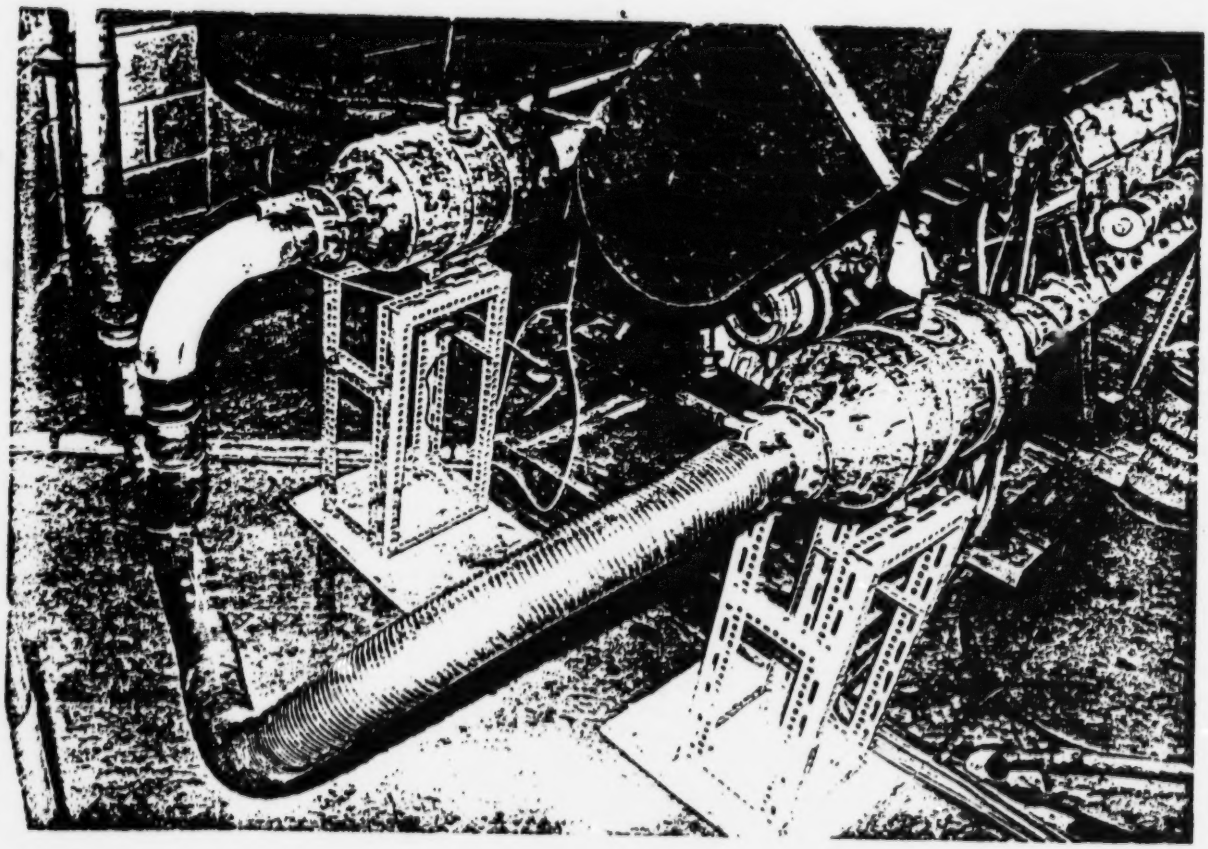


FIGURE 1 - Catalytic Purifier Installation

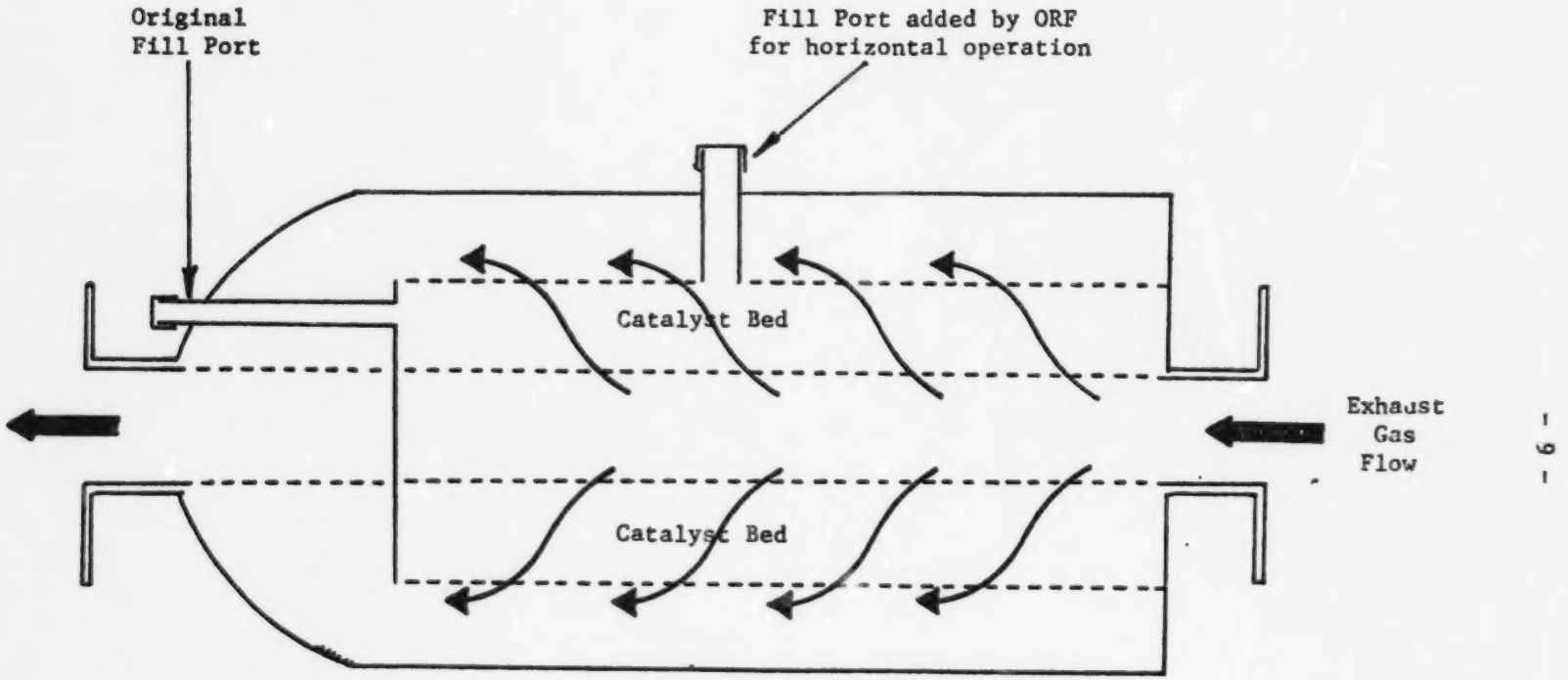


FIGURE 2 - Oxycat Catalytic Purifier

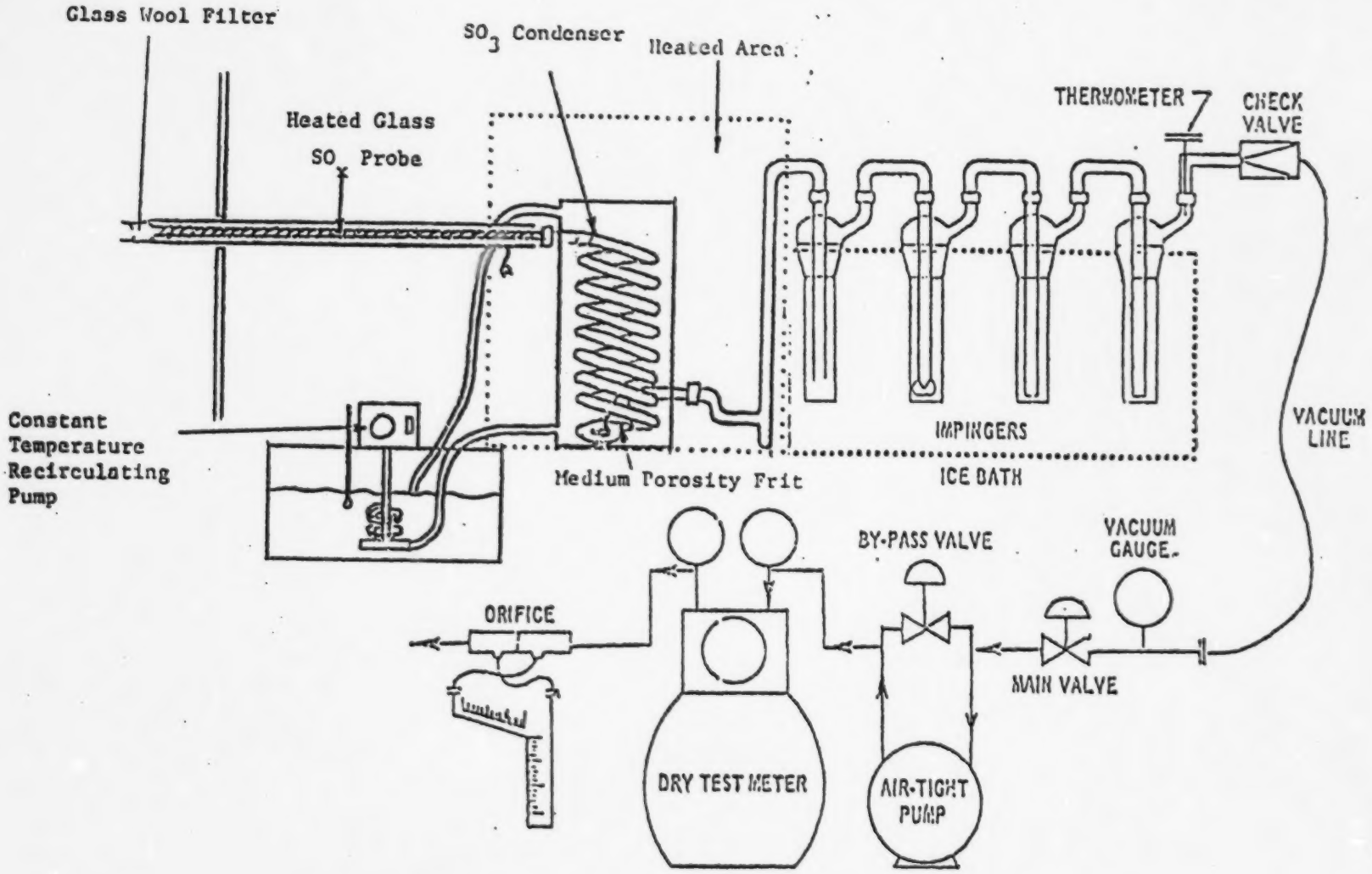
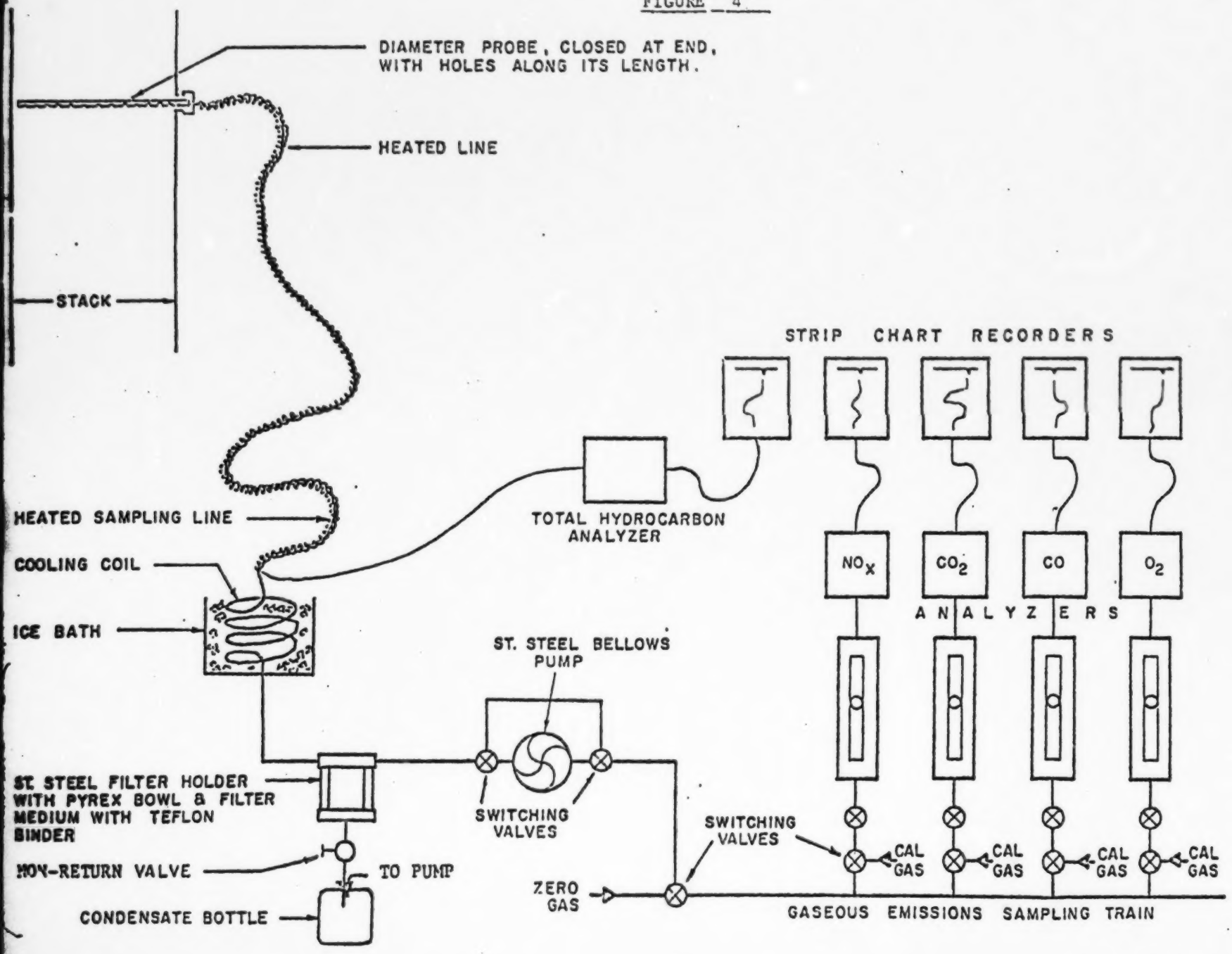


FIGURE 3 - Sampling Train for SO_2/SO_3

FIGURE 4


DIAMETER PROBE, CLOSED AT END, WITH HOLES ALONG ITS LENGTH.
HEATED LINE
STACK
STRIP CHART RECORDERS
HEATED SAMPLING LINE
TOTAL HYDROCARBON ANALYZER
COOLING COIL
NOx
CO2
CO
O2
ANALYZERS
ICE BATH
ST. STEEL BELLOWS PUMP
ST. STEEL FILTER HOLDER WITH PYREX BOWL & FILTER MEDIUM WITH TEFLON BINDER
SWITCHING VALVES
SWITCHING VALVES
NON-RETURN VALVE
TO PUMP
CAL GAS
CAL GAS
CAL GAS
CAL GAS
ZERO GAS
CONDENSATE BOTTLE
GASEOUS EMISSIONS SAMPLING TRAIN

APPENDIX 1

TURBIDIMETRIC METHOD

Procedure

a. Formation of barium sulfate turbidity: Measure 100 ml sample, or a suitable portion made up to 100 ml, into a 250-ml erlenmeyer flask. Add exactly 5.00 ml conditioning reagent and mix in the stirring apparatus. While the solution is being stirred, add a spoonful of $BaCl_2$ crystals and begin timing immediately. Stir for exactly 1 min at a constant speed.

b. Measurement of barium sulfate turbidity: Immediately after the stirring period has ended, pour some of the solution into the absorption cell of the photometer and measure the turbidity at 30-sec intervals for 4 min. Because maximum turbidity usually occurs within 2 min and the readings remain constant thereafter for 3 to 10 min, consider the turbidity to be the maximum reading obtained in the 4-min interval.

c. Preparation of calibration curve: Estimate the sulfate concentration in the sample by comparing the turbidity reading with a calibration curve prepared by carrying sulfate standards through the entire procedure. Space the standards at 5 mg/l increments in the 0- to 40-mg/l sulfate range. Above 40 mg/l the accuracy of the method decreases and the suspensions of barium sulfate lose stability. Check reliability of the calibration curve by running a standard with every three or four unknown samples.

d. Correction for sample color and turbidity: Correct for the color and turbidity present in the original sample by running blanks from which the $BaCl_2$ is withheld.